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CHEMICALLY INDUCED FIRES IN AIRCRAFT ELECTRICAL CIRCUITRY BY CLYCOL/WATER SOLUTIONS. HAZARD ANALYSIS AND ELIMINATION METHODS

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FINAL ENGINEERING REPORT

SECURITY CLASSIFICATION: UNCLASSIFIED

TASK NUMBER MME 74-20-147

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FOREWORD

Investigations and tests were performed during this program to evaluate and find methods of reducing the hazards of glycol induced fires in aircraft electrical circuits. Electrolysis tests, glycol penetration tests of connectors, and contamination and decontamination tests were airplane oriented. Postulation and verification of electrochemical reactions were involved to the extent necessary to characterize the hazards and evaluate counteracting substances.

Glycol immersion tests of components and hundreds of electrolysis tests were required to determine the nature of the hazards and the suitability of preventive measures. Failure mechanisms proved to be more varied and obscure than anticipated. Program schedules did not permit detailed chemical analysis of all the reactions observed. The reactions were evaluated for potential airplane hazard and for effectiveness of inhibiting chemicals.

Chemical and electrolytic tests and analyses were performed by H. J. Weltman of the Chemistry Laboratory. Penetration and immersion tests were performed by A. J. Ledwig of the Electrical Test Laboratory.

ABSTRACT

As a result of airplane electrical connector fires traced to the presence of glycols, investigations have been undertaken to identify the mechanism of the involved hazards and to find methods of counteracting the hazards. Tests were performed with ethylene glycol, propylene glycol, and airplane deicing fluid as electrolytes in a wide range of dilution with water. Silver coated wire, gold coated connector pins, and rhodium coated connector pins were used as electrodes. Electrode spacings were determined which would produce the characteristic smoke and flames when 28 volts direct current was applied to the electrodes and the electrolyte was applied a drop at a time.

Tests were repeated with various additives intended to form non-ionizing reactants or otherwise stop the flow of current or exothermic chemical reaction that resulted from the electrolysis. These tests graded reaction inhibitors according to the range of dilutions and spacings over which they were effective. The candidate solution was tested for compatibility with airplane materials.

Airplane areas subjected to various glycol solutions, contamination detection, and decontamination procedures were investigated. Airplane wiring and connectors were tested for susceptibility to glycol penetration. Component damage was induced to evaluate the failure mechanism.

This report details the tests performed, the variables conducive to a fire hazard and the procedures for reducing the hazard.

TABLE OF CONTENTS

Abbreviations:

SYMBOL	NAME
BZT	Benzotriazole
EMI-24	2-ethyl, 4-methyl imidazole
TOT	sodium salt of tolyltriazole

Definitions:

"Inhibitor" refers to additives to glycol solutions for corrosion control, buffering, or wetting.

"Reaction Inhibitor" refers to additives to glycol solutions to retard electrolytic chemical reaction.

CHEMICALLY INDUCED FIRES IN AIRCRAFT ELECTRICAL CIRCUITRY BY GLYCOL/WATER SOLUTIONS - HAZARD ANALYSIS AND ELIMINATION METHODS

I. INTRODUCTION

The reaction of silver coated wires energized by a direct current electrical circuit in a glycol/water electrolyte was investigated by NASA and reported in References (1), (2), and (3). The reaction was initiated by placing a drop of glycol solution between the energized wires. As electrolysis proceeded, the electrolyte was replenished a drop at a time with several minutes between drops. With proper control of drop rate, electrolysis continued, electrolyte composition changed, and smoke and flames were produced.

The aircraft industry, in general, did not at that time consider this phenomenon a potential hazard on airplanes. Laboratory demonstrations required small scale controlled conditions of drop rate and electrode spacing. These conditions did not appear applicable to airplane wiring methods and glycol solution usage.

Recently, several airplane circuit failures have indicated a need for re-examination of the silver-glycol reactions. Four separate incidents of equipment connector fires and one incident of printed circuit connector failure showed overheating considerably in excess of the energy contributed by the electrical circuit. In each case, the area had previously been wetted by glycol solutions. The failures were significant, both in the loss of circuit function, and in the associated fire hazard.

Further investigation of the failures reinforced the conclusions that a silver-glycol reaction occurred in the equipment connector and probably a gold-glycol reaction occurred in the printed circuit connector. This led to a program of hazard analysis and hazard elimination considering the electrical components and materials that can form electrolytic cells and the glycol solutions that can support the reaction.

Initially the program was envisioned to require hazard analysis of glycol solutions on airplanes, analysis of the probability of glycol induced aircraft fires, identification of susceptible subsystems, and determination of corrective measures in terms of hardware and fluid changes and procedure changes.

In the process of hazard analysis and reaction characterization, it became evident that any subsystem DC circuits exposed to glycol solutions were subject to failure, and that the probability of failure involved the probability of prior installation damage or maintenance error rather than deficient hardware or statistically determinable reaction variables. Exposure of aircraft systems to glycol solutions were found to be extensive. Hazardous reactions were obtained over a wide range of solution types and concentrations.

The test program developed into a reaction survey of glycol solutions, circuit spacing, hardware susceptibility, reaction inhibiting materials, and methods of preventing hazardous reactions.

II. SUMMARY

There are many electrical components, wires, and connectors unavoidably exposed to various contaminants on aircraft. Water, salt spray, runway deicing chemicals, airplane cleaning and deicing fluids are all externally applied potential contaminants. Internal to the airplane are hydraulic fluids, oil, coolant fluids, and fuel that may be circuit contaminants by spillage or leakage. These substances or combinations of them can have damaging effects on electrical circuitry. Electrolysis of conducting fluids, particularly in direct current circuits, causes current leakage, heat, and deplating. Contamination of some conductors by ethylene glycol or propylene glycol solutions adds the hazard of exothermic electrolytic reaction accompanied by smoke and flames.

Aircraft electrical circuit components were designed to be resistant to contamination. Tests show that properly assembled undamaged electrical wiring, connectors, and equipment are highly resistant in this respect. However, there have been glycol induced circuit fires on airplanes. The nature of the failure mechanism and preventive measures were investigated in this program.

Laboratory tests of wires and connectors showed no glycol penetration or electrolytic action in undamaged, assembled wires and connectors. Damaged wire insulation at a connector, or damaged connector insert material could permit destructive glycol reactions. Glycol solutions introduced between the plug and receptacle prior to mating could penetrate to the contact support surface and cause internal burning between positive and negative 28 volt dc energized contacts.

Connector or wire damage or mishandling can permit penetration of contaminants between conductors. If the contaminant is a glycol solution, energized 28 volt dc conductors can trigger a violent reaction and fire. This possibility suggests that, in addition to avoiding contamination or circuit damage where possible and decontaminating where necessary, the glycol reaction itself should be stopped.

Of the many chemicals tested for their ability to inhibit the reaction, benzotriazole, tolyltriazole, and 2-ethyl, 4-methyl imidazole were found to have inhibiting properties. Benzotriazole is most effective. The other chemicals could be considered if benzotriazole is found to be too hazardous to personnel.

With reasonable quantities of reaction inhibitor, the glycol electrolytic reaction is not completely neutralized for all solution dilutions and electrode spacings possible. For each type of glycol solution used on airplanes, there is an optimum range of reaction inhibitor quantity that will prevent hazardous reactions under practical conditions and still not leave excessive amounts of residue in contaminated areas.

Glycol solutions in concentrations below 1% did not react under any test condition. This can be applied to glycol decontamination in the development of washing and drying procedures compatible with each area and equipment involved.

III. CONCLUSIONS

The test program revealed no single cure-all for glycol contamination. The hazards of chemically induced fires in aircraft electrical circuitry can be greatly reduced by maintaining the integrity of the insulation and sealing of wiring, connectors, and equipment; by protecting electrical connectors and equipment from glycol solutions with suitable covers during deicing; and by replacing any electrical connectors suspected of glycol contamination while unmated. Normally unmated connectors should be of a self-sealing design or have sealing covers if they can be subjected to glycol solutions.

Glycol wetted areas should be inspected for frayed insulation, damaged connectors, missing connector filler plugs, open connectors, and open electrical equipment. Repairs should be made prior to glycol exposure. Electrical circuitry inadvertently exposed to glycol solutions should be rinsed and dried using procedures compatible with the equipment involved.

Benzotriazole can be added to glycol solutions to reduce the possibility of a hazardous glycol reaction in electrical circuitry. Sodium salt of tolyltriazole or 2-ethyl, 4-methyl imidazole also have reaction inhibiting qualities but they are less effective than benzotriazole. None of the reaction inhibitors tested completely neutralize electrolysis for all conditions. However, reaction inhibitor quantities suitable for silver are more than adequate for gold or rhodium plating.

It is recommended that further investigations be made prior to decisions regarding the use of reaction inhibitors:

1. Study the National Cancer Society's evaluation of potential reaction inhibitors as carcinogens (information expected in 1975).

2. Standardize on an ethylene glycol solution with corrosion inhibitors suitable for aircraft and determine compatible quantities of reaction inhibitor.

3. Investigate non-ionizing corrosion inhibitors as a replacement for potassium phosphate in standard deicing fluid as a means of reducing reaction inhibitor quantity.

4. Conduct analytical investigation of glycol electrolytic reactions with other metals commonly used in electrical circuits. It is suspected that present cautions regarding "silver-glycol" hazards should be extended to other metals. Hopefully, the same reaction inhibitors effective for silver, gold, and rhodium platings will be effective for other reacting metals.

IV. DISCUSSION

A. Glycol Induced Airplane Circuit Failures

Airplane electrical circuit failures attributed to glycol exposure had several features in common. All of the failures were in electrical circuits continuously energized at 28 volts dc. All of the failed connectors were subjected to exposure to glycol solutions. The failed connector that was analyzed showed evidence of local internal high temperature with burned wire insulation and connector insert material. Metal pins and sockets were partially melted. However, the wiring a short distance from the connector was undamaged, circuit breakers were not tripped, and there was no evidence of an electrical overload. Analysis of the failed connector by infrared spectrometry showed the presence of propylene glycol.

All the evidence indicated that electrolysis of the glycol solution by direct current in the milliampere range had induced high temperatures, smoke, and flames in the manner reported by NASA for a silver-glycol electrolytic reaction. Figure 1 shows the damage to a connector and wiring that occurred on an airplane.

B. Electrolytic Reactions

Electrolysis of the glycol/water solutions in a dc circuit results in the formation of hydrogen at the cathode and some form of oxidation at the anode. The cathode tends to brighten by reduction and the anode tends to oxidize and form a black residue. In general, the chemical changes and rate of change are dictated by electrode size and spacing; electrolyte dilution, volume, and temperature; source voltage and impedance; and the presence of additives in the solution.

The silver electrode - glycol/water electrolyte reaction described by NASA involved the formation of silver oxide at the anode. The silver oxide reacted to dehydrate the ethylene glycol. Decomposition products of ethylene glycol together with hydrogen at the cathode resulted in the formation of steam, smoke, heat, and flames. Other chemicals in the glycol/water solution resulted in additional reaction products and additional energy release (Reference 1).

Fig. 1 PHOTOGRAPH OF AN ELECTRICAL CONNECTOR AND WIRING REMOVED FROM AN AIRPLANE

The tests performed to evaluate airplane hazards associated with glycol reactions differed from NASA tests in several respects:

<u>Electrodes</u>

Silver plated copper wire, gold plated copper alloy pins, and rhodium plated copper alloy pins were tested at various separations.

<u>Electrolytes</u>

Ethylene glycol/water, propylene glycol/water, and deicing fluid (mixture of ethylene and propylene glycols with additives) solutions were used with a wide range of dilutions.

<u>Power Source</u>

A low impedance (high capacity) 28 volt dc power source was used.

These aircraft related differences may have caused chemical changes and reaction products differing from the general type of reaction described by NASA. Since this program is concerned with identifying and preventing hazardous glycol reactions in electrical circuits, the exact nature of the reaction is of concern only to the extent that it indicates a method for preventing the reaction.

C. Reaction Inhibitors

NASA studies indicated that benzotriazole (BZT) effectively prevents the silver electrode-ethylene glycol/water electrolyte reaction. The program for aircraft hazard analysis included benzotriazole and other candidate reaction inhibitors. These chemicals were selected as silver chelating agents, as chemicals that may form insoluble substances (such as sulfides), or as chemicals that might form other reaction products which inhibit the electrolytic action. The substance that was most effective in retarding the glycol reaction (reaction inhibitor) was further checked for compatibility with airplane materials and suitability for handling by ground crews (toxicity).

D. Glycols

A mixture of ethylene glycol and propylene glycol together with corrosion inhibitors, a wetting agent, and water is used extensively in deicing and anti-icing airplane exterior surfaces (Reference 5). Wing, tail, and landing gear areas are all wetted with the solution. Associated areas are subject to glycol wetting by splashing or runoff.

Many military airplanes use propylene glycol in water boilers and air pressurization system anti-icing. The low toxicity of propylene glycol makes it ideal for any systems associated with the environmental control systems for the flight crew. Ethylene glycol is preferred for liquid cooling loops in the cooling of avionic equipment. The various glycol solutions and dilutions were investigated in this program.

E. Electrode Materials

Airplane electrical wiring may be tin coated copper, silver coated copper, or nickel coated copper. In smaller gages, copper alloys with silver or nickel coating may be used in lieu of copper. Electrical connectors use copper alloy or stainless steel contacts plated with silver, gold, or rhodium. Electrical components (switches, circuit breakers, relays, etc.) may use any or all of the above metals. All of these metals and others are potential electrodes.

NASA tests indicated that copper, tin coated copper, and nickel coated copper do not produce an exothermic electrolytic reaction. Therefore, this program did not involve those materials specifically. It did involve testing silver plated copper wires and the various metals used in connector contacts.

F. Test Program Objectives

This program is concerned with investigations of the electrolytic activity and hazards associated with the various glycol solutions and with evaluation of methods of reducing the hazards. It includes a survey of glycol solutions, glycol reactions, glycol penetration of electrical circuits, contamination detection, decontamination methods, and reaction inhibiting methods.

V. Glycol Usage on Airplanes

The most widely used glycol/water solution for airplanes is MIL-A-8243B(2) anti-icing and deicing-defrosting fluid. This fluid is composed (by weight) of a minimum of 88% ethylene and propylene glycol in a 3 to 1 ratio respectively, 0.9 to 1.1% dibasic potassium phosphate, and 0.45 to 0.55% sodium di - (2-ethylhexyl) sulfosuccinate. This fluid is sprayed on the airplanes either at full strength or at various dilutions down to 20% fluid and 80% water (by volume). Further dilution occurs as the fluid mixes with melting ice or snow. Reference 5 details application procedures. MIL-A-8243B deicing fluid is also used as a low temperature rinsing material following the application of MIL-C-27251 cleaning compound. Reference 8 describes application procedures.

In addition to the deicing fluid mix of ethylene glycol and propylene glycol, water boilers of some airplane environmental control systems use propylene glycol. These solutions are usually 5 to 10% propylene glycol in water without additives. Some air pressurization systems use 100% propylene glycol fed through the system as a single pass anti-icing measure. Both of these applications involve systems that interface with the air conditioning system for the flight crew. Propylene glycol is used because of its low toxicity.

The properties of ethylene glycol make it a preferred heat transfer medium in liquid cooling loops. High density, high power avionic systems are making the use of this type of cooling system more attractive. Increased usage of ethylene glycol on military airplanes is anticipated.

A standard ethylene glycol coolant formulation has not been established for aircraft. Probably eutectic mixtures of about 62% ethylene glycol (by weight) in water with additives will be selected based upon compositions developed by NASA (References 1, 2, and 3).

All of these glycol solutions are very active electrolytes and produce the glycol exothermic reaction. Of lesser importance is low temperature aircraft surface cleaning compound MIL-C-27251A containing 0.95% ethylene glycol by weight, and alkaline waterbase aircraft surface cleaning compound MIL-C-25769G(1) containing 5.1% (by weight) ethylene glycol, n-mono butyl ether.

Table I summarizes glycol usage on airplanes. Deicing fluid is used routinely in large quantities over wide areas of airplanes. Opportunities for equipment contamination are numerous and frequent. Propylene glycol contamination has been encountered by water boiler overflow and by seal rupture between the fluid and cooling air ducts. Seal rupture has permitted the glycol to enter forced air cooled equipment remotely located from the water boiler. Similar failures could occur with ethylene glycol cooling loops.

Table I. Glycol Solutions Used on Airplanes

GLYCOL FLUID % BY WEIGHT	ADDITIVES	APPLICATION	PROBABLE AREAS OF CIRCUIT CONTAMINATION
ETHYLENE GLYCOL 62% APPROX.	CORROSION INHIBITORS - NO PRESENT STANDARD FOR AIRCRAFT	LIQUID COOLING SYSTEM	o HEAT EXCHANGER AREAS o FILL AREAS o PLUMBING FITTING AREAS o EQUIPMENT USING FORCED AIR COOLING WITH A LIQUID/AIR HEAT EXCHANGER
PROPYLENE GLYCOL 100%	NONE	AIR PRESSURIZATION SYSTEM ANTI-ICING	o FILL AREAS o PLUMBING FITTING AREAS
PROPYLENE GLYCOL 10% APPROX.		WATER BOILERS	o WATER BOILER AREA o FILL AREA o PLUMBING AND FITTING AREAS o EQUIPMENT USING FORCED AIR COOLING WITH A LIQUID/AIR HEAT EXCHANGER
PROPYLENE GLYCOL 5% APPROX.		WATER BOILER REFILL	
ETHYLENE GLYCOL AND PROPYLENE GLYCOL (3:1 RATIO) 88% MIN. MIL-A-8243B(2)	CORROSION INHIBITORS AND WETTING AGENT	ANTI-ICING & DEICING AIRPLANE EXTERIOR SURFACES	o WING, TAIL, FUSELAGE, AND WHEEL WELL AREAS o SEEPAGE & RUN-OFF AREAS o PYLONS o EXTERNAL STORES
ETHYLENE GLYCOL 0.95% (LOW TEMPERATURE CLEANING COMPOUND MIL-C-27251A)	CLEANING COMPOUND	CLEANING AIRPLANE EXTERIOR SURFACES	
ETHYLENE GLYCOL, n-MONO BUTYL ETHER 5.1% (ALKALINE WATERBASE CLEANING COMPOUND MIL-C-25769G(1))			

VI. Exploratory Tests

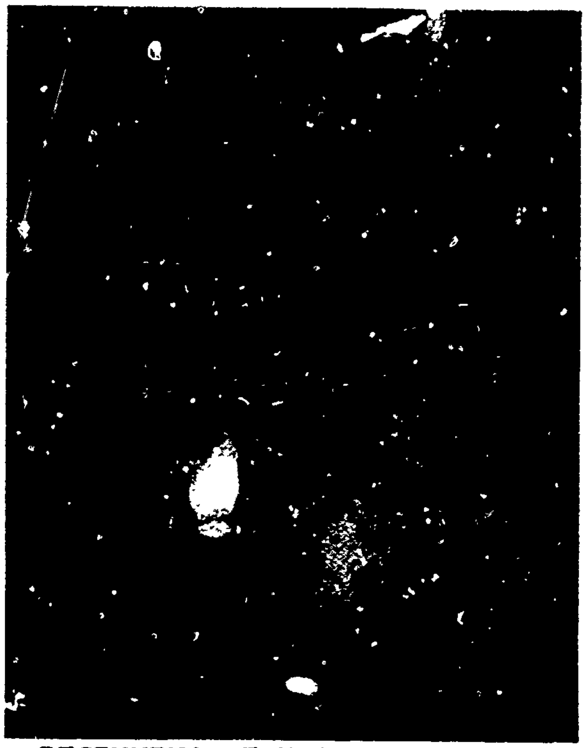
Initial testing attempted to duplicate the observed connector fires. Failures could not be induced by applying glycol solutions to properly assembled connectors. Eventually, a glycol reaction was induced by removing the wire insulation at the connector insert and tying the bare wires closely together. Glycol solution was applied drop by drop between the bare wires. The sequence of events are shown in Figure 2. Further tests of glycol penetration possibilities were conducted during the program.

Exploratory tests of silver coated wires showed that ethylene glycol, propylene glycol, and MIL-A-8243B deicing fluid would all react in a 28 volt dc circuit to produce smoke and flames. The reaction was sensitive to wire spacing, wire size, and solution dilution.

Careful observation was required in marginal cases to identify a true glycol reaction. At close spacing, distilled water alone would gradually form a residue and emit small sparks and radio noise. This was the so-called "wet fire" phenomenon (Reference 1, page 4) and is of interest to this program only if a fire hazard is involved. Unplated copper wires closely spaced in a glycol solution also form a residue and exhibit some of the characteristics of a silver-glycol reaction. This may have been a "wet fire" fueled by glycol.

Tests with silver-glycol reactions showed the production of copious quantities of smoke and flames characteristic of the reaction. The type of reaction was assessed by the experimenter based upon experience gained from extensive testing. Radio noise and ammeter variations were monitored to indicate the level of activity.

The tests indicated that a standard test configuration for an electrolytic cell was needed to compare reaction variables and to determine the effectiveness of various reaction inhibitors.



BEGINNING OF FLAME ERUPTION

FLAME PROPAGATES TO THE WIRE INSULATION

CONNECTOR INSERT AND WIRE INSULATION IGNITED

Fig. 2 LABORATORY INDUCED CONNECTOR FIRE

VII. Reaction Characterization and Reaction Inhibitor Evaluation

A. Test Cell

All reaction tests used standard test cell configurations consisting of 3 by 5 inch sheets of cardboard with the electrodes secured with masking tape and adjusted for the desired spacing between electrodes. The arrangement for silver plated copper wire and for gold or rhodium connector pins are shown in Figure 3. The cathode was #12 AWG bare solid copper wire. This permitted closer dimensional control of the electrodes, added rigidity to the assembly, and had no effect on the reaction products. The silver plated copper wire was stranded 12 AWG. Spacings between electrodes were 0.005, 0.010, 0.015, 0.020, 0.040, 0.080, and 0.100 inches. New test cells were used for each test reaction and each test spacing.

B. Test Procedure

The electrodes were connected to a 28 volt direct current power source. Initially, a dry cell battery was used. This was not considered representative of airplane 28 volt systems in capacity or internal impedance. The battery source was replaced by the laboratory 28 volt dc power system early in the program.

Current was measured with a shunt and digital meter. Time was recorded from the time of application of the electrolyte to the onset of the exothermic reaction as evidenced by smoke and flames.

The electrolyte was applied a drop at a time between electrodes. Drop rate was determined by noting the electrolytic reaction through a low power microscope, the variations of the digital ammeter, and the noise of the portable radio (when used).

Individual reaction tests were performed for the various solutions, dilutions, reaction inhibitors, and electrode spacings under study.

C. Reaction Characterization

A series of tests were performed to identify the variables involved in the reaction:

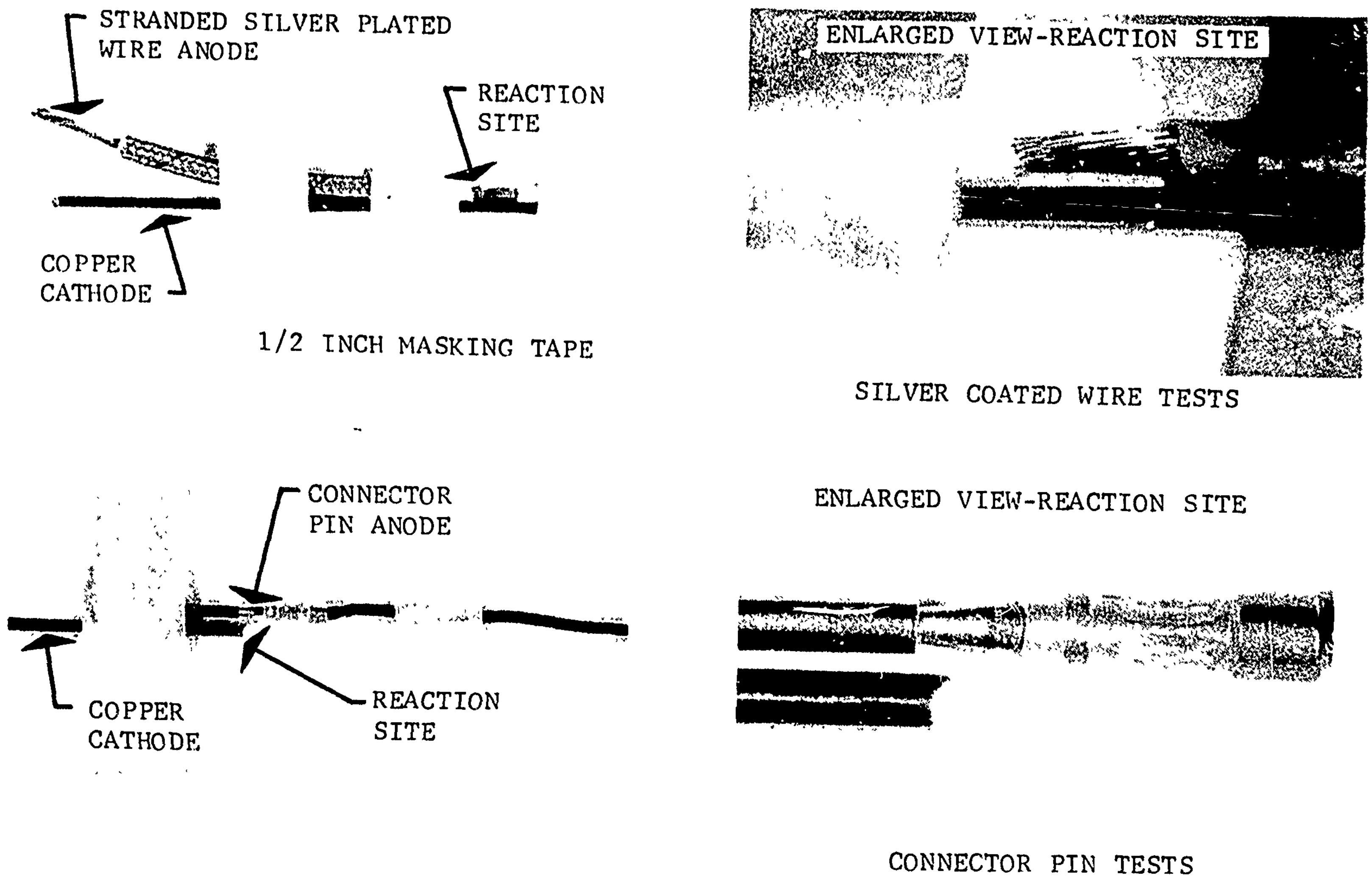


Fig. 3 GLYCOL REACTION TEST CONFIGURATIONS

1. Conductors (Electrodes)

Silver coated copper wire, gold coated connector pins, and rhodium coated connector pins with spacings of from .005 to 0.10 inches were used as anodes.

2. Solutions (Electrolytes)

Ethylene glycol (100%), propylene glycol (100%), deicing fluid (MIL-A-8243B), and water boiler fluid (5% propylene glycol in water) were the basic test fluids. These solutions were tested in concentrations of 100% to 10% in 10% steps plus 5% and 1% test solutions in water.

The reaction for each spacing, test fluid, solution dilution, and electrode material was observed and the time required to produce smoke and flames was recorded.

D. Reaction Inhibitor Evaluation

With the behavior of glycol solutions defined, the next step was to screen potential reaction inhibitor chemicals to evaluate their ability to impede the reaction. The following substances were tested:

Item No.	Chemical
1	Acetic acid
2	Benzotriazole (BZT)
3	Dextrose
4	Dimethyl sulfoxide
5	Diphenyl phosphate
6	Ethylenediaminetetraacetic acid (Versene acid)
7	Glucose
8	Glycerin
9	Hydroquinine
10	Sodium polysulfide
11	Sucrose
12	Stearic acid
13	Tetrasodium salt of Ethylenediaminetetraacetic acid (Versene)
14	Thiourea
15	Sodium salt of Tolyltriazole (TOT)
16	Versene Fe-3

17	1 methyl imidazole
18	2-ethyl, 4-methyl imidazole (EMI-24)

Of these substances, only Items 2, 15, and 18 showed significant retarding reactions. Characterization tests of reaction inhibitor quantity, glycol solution dilution, and electrode spacing were run for these three candidate chemicals.

Ideally, the reaction inhibitor would completely neutralize the glycol exothermic reaction for all solution dilutions and electrode spacings. Glycol solution concentrations normally used are discussed in Section V. It is assumed that mixing with rain, snow, or ice could provide any dilution and evaporation could provide essentially any concentration of the glycol solutions. Electrode spacing could vary from a very large distance down to essentially zero spacing. Localized insulation damage of airframe wires without compacting or wire to wire abraiding would give wire-to-wire spacings of about 0.015 inches. Connector minimum internal metal to metal spacing is 0.022 inches. Damaged wire bundles could give any metal to metal spacing down to a short circuit. Actually, relatively large quantities of reaction inhibitor were required to prevent the glycol reaction for all solution dilutions at 0.005 inch spacing between electrodes. The data obtained permits a comparison of inhibiting properties versus reaction inhibitor quantity for the best practical balance.

The standard test configuration used provides a basis of comparison. The drop by drop testing does not represent the usual contamination condition on airplanes. Testing with larger quantities of fluid "floods" the reaction. A residue and smoke are formed but flames do not occur until the quantity of fluid reduces by evaporation, boiling, and electrolysis. The standard test cell approach is believed to be a valid method of reaction evaluation.

E. Reaction Test Results

The results of reaction tests and reaction inhibitor tests are shown in Table II and summarized in Graphs I, II, and III.

1. Ethylene Glycol

Ethylene glycol reacts with silver plated wire up to a maximum electrode spacing of 0.080 inches. Table II shows that benzotriazole (BZT) reduces the maximum reacting spacing particularly at the higher fluid concentrations. EMI-24 has inhibiting effects much less pronounced than those of BZT.

Sodium salt of tolyltriazole (TOT) was more effective than EMI-24 but less effective than BZT.

Gold plated electrodes reacted up to 0.020 inches at 5% to 10% concentrations and up to 0.015 inches at 5% to 40% concentrations. With the addition of 1 gram of BZT per 100 ml of test fluid, no reaction was obtained over the range tested (0.015 inches minimum). Rhodium plated electrodes were similar to gold but reactive over slightly wider concentration range and were not completely inactive with 1 gram of BZT. It was concluded that gold or rhodium plated pins were much less active than silver plated wire and that reaction inhibitor quantities suitable for silver plated wire were more than adequate for gold or rhodium plating.

2. Propylene Glycol

Propylene glycol was less active than ethylene glycol and reaction inhibitors were more effective in reducing the reaction. BZT was the most effective.

3. Deicing Fluid (MIL-A-8243B)

Deicing Fluid was somewhat less active than ethylene glycol. However, this fluid is used with a wide range of dilutions and it was difficult to inhibit reactions at the lower concentrations. A wide range of reaction inhibitor quantities and combinations were tested as shown in Table II.

4. Synthetic Deicing Fluid

Indications were that the corrosion inhibitors in MIL-A-8243B deicing fluid increased electrolytic activity of the solution and that the increased activity prevented the accumulation of the non-ionizing reaction inhibiting gel around the anode. As a result, larger quantities of reaction inhibitor were required to increase the rate of formation of the insulating gel. It was believed that the use of non-ionizing corrosion inhibitors would permit a reduction in reaction inhibitor quantity.

To test this premise, a glycol solution was prepared similar to MIL-A-8243B fluid but without the additives. This "synthetic" deicing fluid was tested as shown in Table II. The synthetic deicing fluid without reaction inhibitors was more active than MIL-A-8243B fluid indicating that the corrosion inhibitor phosphates had some retarding effects plus increased electrolysis. Phosphates, however, did not prove to be good reaction inhibitors.

The addition of BZT to synthetic deicing fluid proved more effective than similar quantities in MIL-A-8243B fluids.

5. Water Boiler Fluid (5% propylene glycol)

Tests on water boiler fluid were performed using small increments of reaction inhibitor to determine acceptable effective usage. Test results are shown in Table II.

6. Effects of Sodium Chloride

There was some concern that salt water might neutralize the effects of BZT. The tests summarized in Table II show these effects to be negligible. The salt creates two opposing conditions. Conductivity of the electrolyte is increased and the formation of chlorides by the reaction reduces electrical conductivity at the silver anode.

7. Cleaning Compound MIL-C-27251A

This compound does not contain enough glycol to cause a reaction.

8. Cleaning Compound MIL-C-25769G

The ethylene glycol n-mono butyl ether of this compound does not exhibit the exothermic glycol reaction. The compound is an active electrolyte but it dissipates without smoke or flames.

F. Optimum Reaction Inhibitor

There are many variables involved in determining whether or not an exothermic glycol reaction will occur. The glycol solution may evaporate and electrolyze away before sufficient residue is formed to cause the reaction. There may be so much glycol solution present that the residue is formed but the reaction cannot occur. Spacing between conductors may be too large for the solution concentration to react. The voltage between conductors may be too low for reaction or high enough to react at abnormally large spacings. (This program was concerned with 28 volt dc source voltages.)

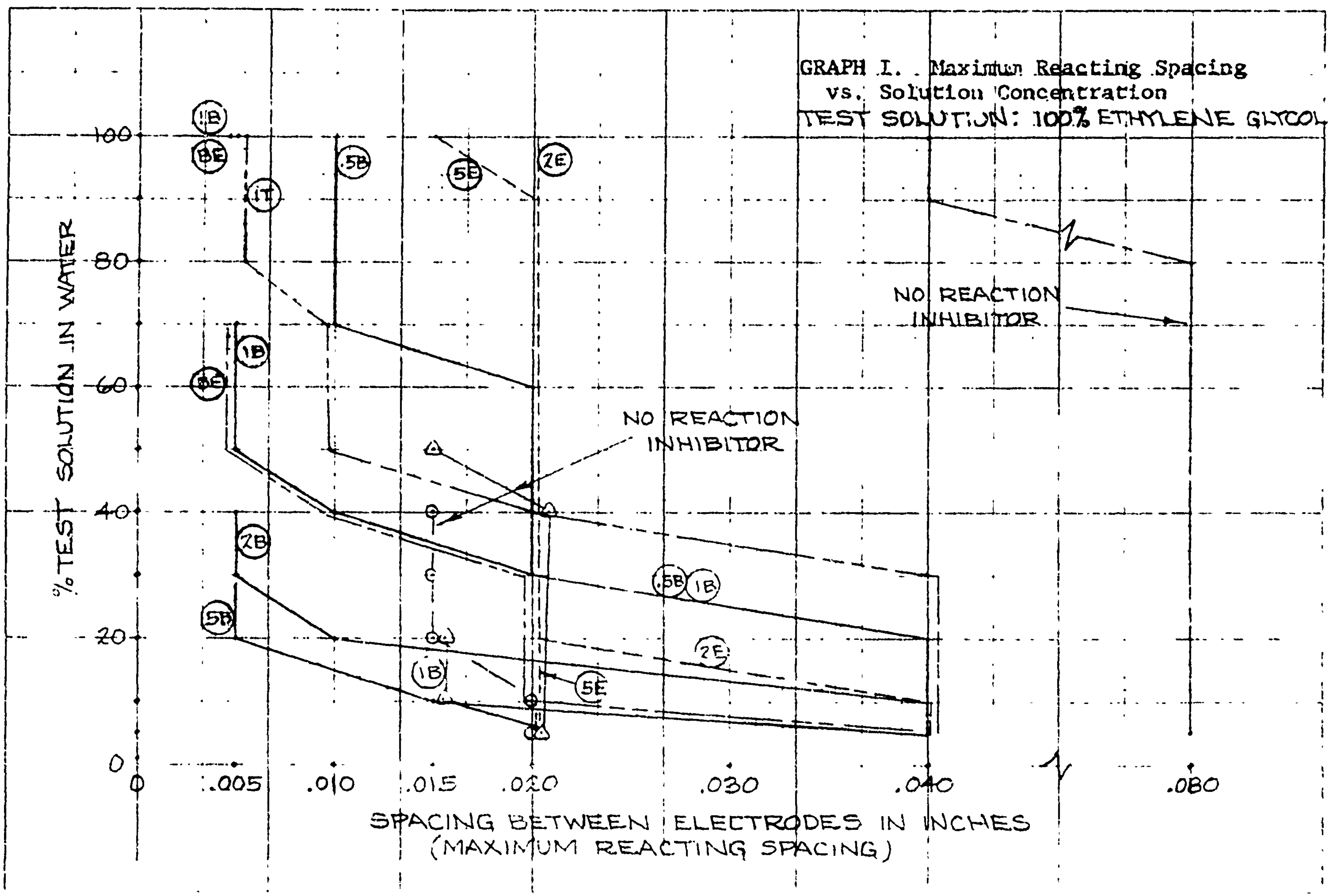
For airplane 28 volt dc circuits, the reaction inhibitor selected must at least prevent the glycol reaction at 0.015 inch spacing between conductors with glycol solutions in the range of concentrations likely to be encountered. Examination of Table II and Graphs I, II, and III shows the preferred minimum quantities of reaction inhibitor summarized in Table III.

SYMBOLS FOR GRAPHS I, II, AND III

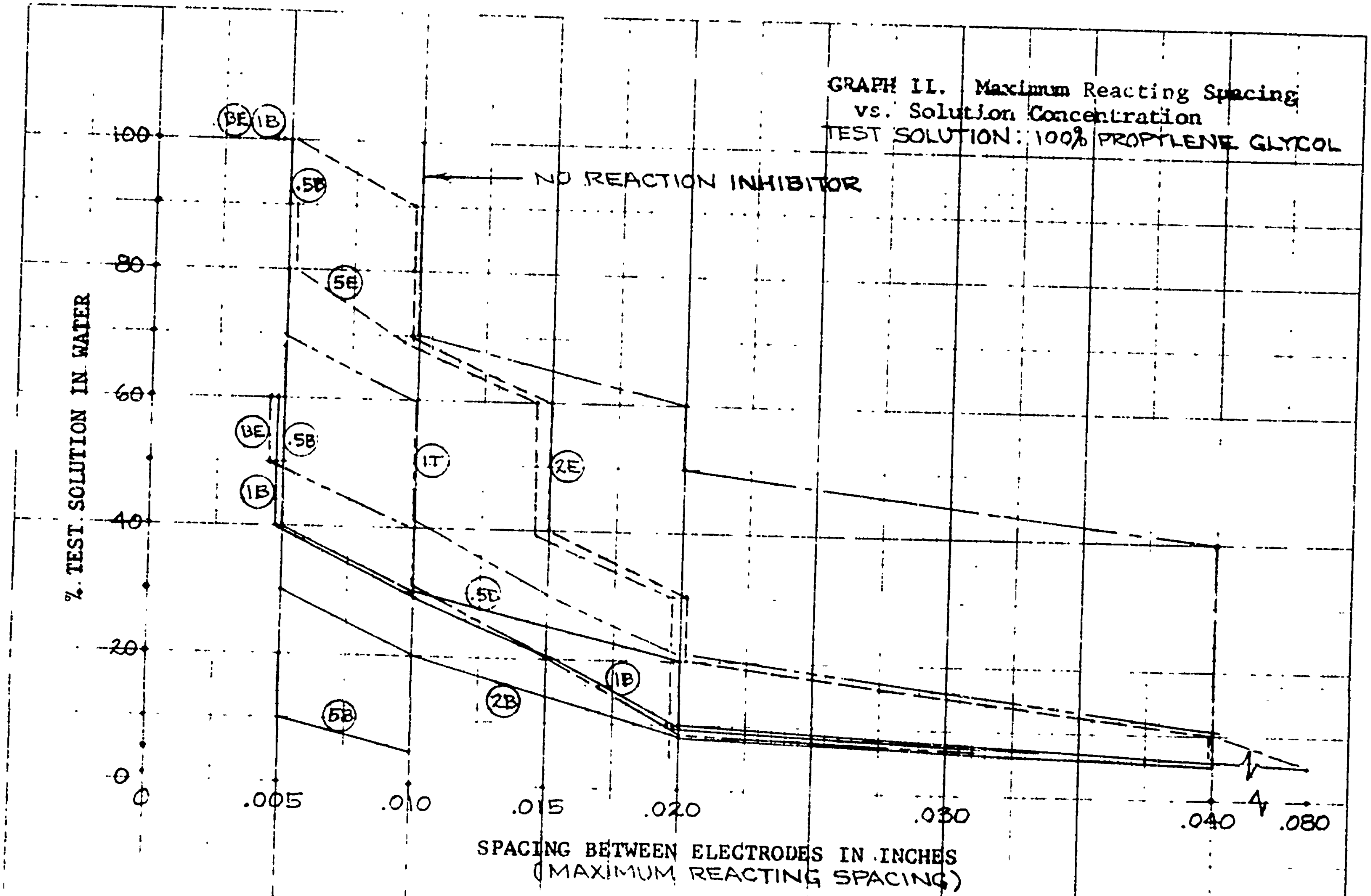
	without reaction inhibitors
	with BZT reaction inhibitor
	with EMI-24 reaction inhibitor
	with BZT and EMI-24
	with sodium salt of tolyltriazole reaction inhibitor (TOT)
	gold plated electrode
	rhodium plated electrode
2B	2 grams BZT per 100 ml of test solution
2E	2 grams EMI-24 per 100 ml of test solution
1T	1 gram TOT per 100 ml of test solution
BE	1 gram BZT and 2 grams EMI-24 per 100 ml of test solution
BE	5 grams BZT and 5 grams EMI-24 per 100 ml of test solution

silver plated wire

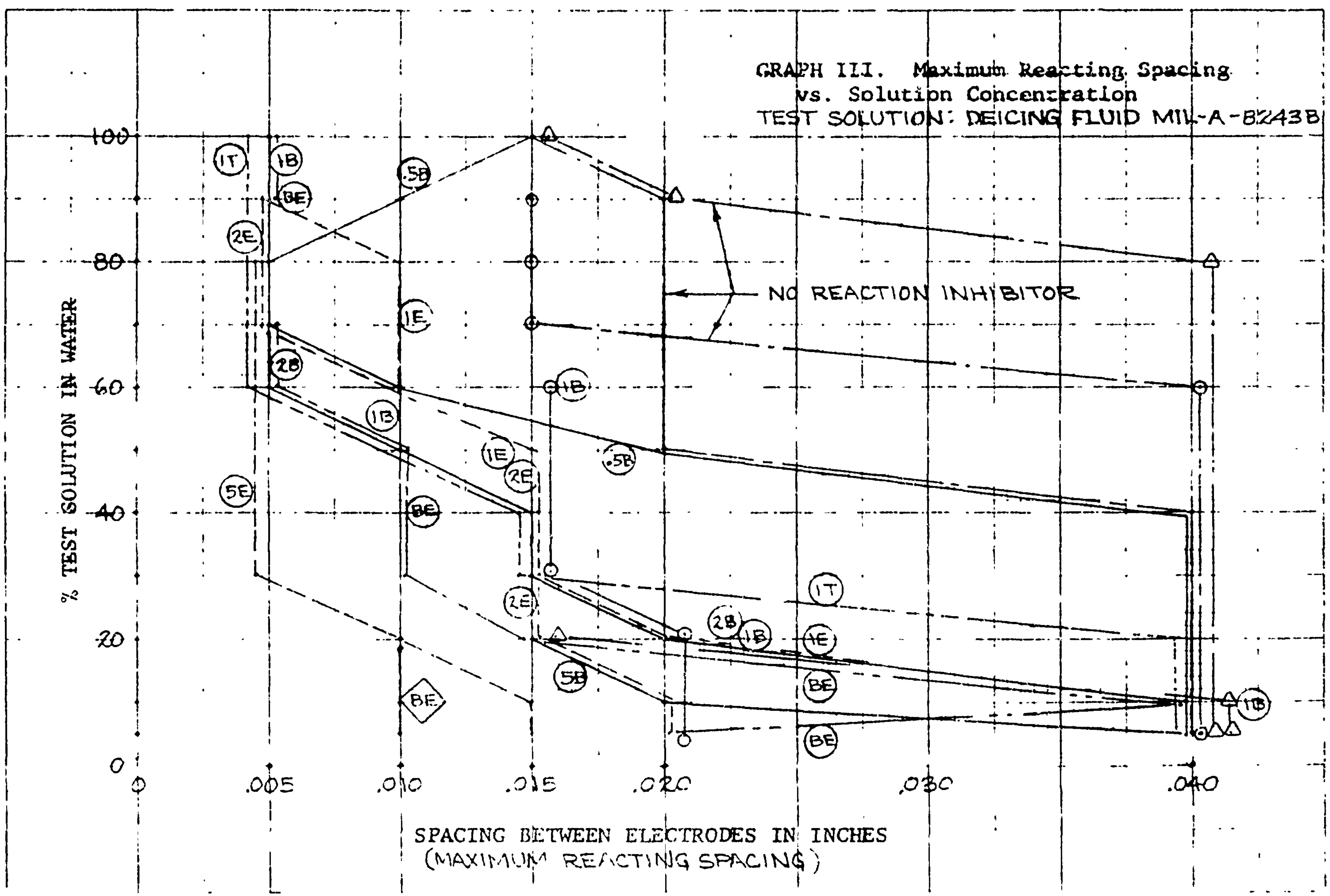
Note: The number in the circle refers to the number of grams of reaction inhibitor per 100 ml of test solution. The letters refer to reaction inhibitors: B for BZT, E for EMI-24, and T for TOT.



GRAPH I. Maximum Reacting Spacing vs. Solution Concentration
TEST SOLUTION: 100% ETHYLENE GLYCOL



GRAPH II. Maximum Reacting Spacing vs. Solution Concentration
TEST SOLUTION: 100% PROPYLENE GLYCOL


GRAPH III. Maximum Reacting Spacing
vs. Solution Concentration
TEST SOLUTION: DEICING FLUID MIL-A-8243B
NO REACTION INHIBITOR
% TEST SOLUTION IN WATER
100
80
60
40
20
0
0
.005
.010
.015
.020
.030
.040
SPACING BETWEEN ELECTRODES IN INCHES
(MAXIMUM REACTING SPACING)
1T
1B
BE
.5B
2E
1E
2B
5E
.5B
2E
5B
1B
1E
BE

List of Tests in Table II

Test Fluid	Anode	Inhibitor	Page
Ethylene Glycol	Silver	None ½g BZT 1g BZT 2g BZT 5g BZT	29
		2g EMI-24 5g EMI-24 1g BZT, 2g EMI-24 1g TOT	30
	Gold	None 1g BZT	31
	Rhodium	None 1g BZT	
Propylene Glycol	Silver	None ½g BZT 1g BZT 2g BZT 5g BZT	32
		2g EMI-24 5g EMI-24 1g BZT, 2g EMI-24 1g TOT	33
Deicing Fluid MIL-A-8243B	Silver	None ½g BZT 1g BZT 2g BZT 5g BZT	34
		1g EMI-24 2g EMI-24 5g EMI-24 1g BZT, 2g EMI-24 5g BZT, 5g EMI-24	35
		1g TOT	36
	Gold	None 1g BZT	
	Rhodium	None 1g BZT	

Test Fluid	Anode	Inhibitor	Page
Synthetic Deicing Fluid (3 parts ethylene glycol, 1 part propylene glycol diluted with 10% water)	Silver	None ½g BZT 1g BZT 2g BZT	37
Water Boiler Fluid (5% propylene glycol)		See Charts	38
Sodium Chloride added to: Ethylene Glycol Propylene Glycol Deicing Fluid	Silver	See Charts	38

Table II Notes:

1. Reaction inhibitor quantities are in grams per 100 milliliters of test solution (prior to dilution).

2. No visible chemical reactions were obtained other than simple electrolysis for:

   a. 1% test solution concentration
   b. Electrode spacings of 0.100 inches.

3. A test is considered non-reacting if smoke and small flames are not produced within 2 hours. Reaction time is measured from the start of the test to the first visible generation of smoke or flames.

4. "Synthetic deicing fluid" is a mixture of ethylene glycol, propylene glycol, and water in the same proportions as in MIL-A-8243B deicing fluid but with all other additives omitted. It is used to simulate a deicing fluid with non-ionizing corrosion inhibitors.

TABLE II. TEST CELL REACTION DATA

TEST FLUID: ETHYLENE GLYCOL			ANODE: SILVER PLATED						
REACTION INHIBITOR	% TEST FLUID	% ADDED WATER	SPACE BETWEEN ELECTRODES (INCHES), *TIME (MINUTES) .005"	.010"	.015"	.020"	.040"	.080"	
NONE	100	0					47		
	90	10		1 TO 10 MINUTES					
	80	20							
	70	30							
	60	40							
	50	50							NO REACTION
	40	60		LESS THAN 1 MINUTE					
	30	70							
	20	80							
	10	90							
	5	95							
1/2g BZT	100	0	17	27					
	90	10	21	30					
	80	20	34	40			NO REACTION		
	70	30		16					
	60	40	1 TO 10		30	90			
	50	50	MINUTES		15	75			
	40	60				30			
	30	70	LESS THAN			15			
	20	80	1 MINUTE				30		
	10	90					15		
	5	95							
1g BZT	100	0	90						
	90	10							
	80	20							
	70	30	30				NO REACTION		
	60	40	25						
	50	50	18						
	40	60		25					
	30	70	1 TO 10	16	14	22			
	20	80		11		20	33		
	10	90					15		
	5	95	< 1	1 TO 10 MINUTES					
2g BZT	100	0							
	90	10							
	80	20							
	70	30							
	60	40					NO REACTION		
	50	50							
	40	60	60						
	30	70	45						
	20	80	15	20					
	10	90					11		
	5	95		1 TO 10 MINUTES					
5g BZT	100	0							
	90	10							
	80	20							
	70	30			NO REACTION				
	60	40							
	50	50							
	40	60							
	30	70	10						
	20	80							
	10	90	< 1	5	12				
	5	95			3	5	20		

* TIME IN MINUTES (TYPICAL)

TEST FLUID: ETHYLENE GLYCOL			ANODE: SILVER PLATED					
REACTION INHIBITOR	% TEST FLUID	% ADDED WATER	SPACE BETWEEN ELECTRODES (INCHES), TIME (MINUTES)					
			.005"	.010"	.015"	.020"	.040"	.080"
	100	0	5	10	30	25		
	90	10						
	80	20						
	70	30					NO REACTION	
	60	40	TIME NOT					
2g	50	50	RECORDED		15	10		
EMI-24	40	60						
	30	70						
	20	80						
	10	90			1	1	40	
	5	95						
	100	0	3	17	20			
	90	10				18		
	80	20		10	11			
	70	30				30		
	60	40						
5g	50	50	TIME NOT			8	NO REACTION	
EMI-24	40	60	RECORDED					
	30	70						
	20	80						
	10	90						
	5	95						
	100	0	45					
	90	10						
	80	20						
1g	70	30	20			NO REACTION		
BZT	60	40	16					
&	50	50	11					
2g	40	60		30				
EMI-24	30	70	1 TO 10	18	40	90		
	20	80		14	20	30		
	10	90						
	5	95	< 1	1 TO 10 MINUTES			11	
	100	0	75					
	90	10	40					
	80	20	30				NO REACTION	
	70	30		35				
	60	40						
1g	50	50		7				
TOT	40	60			7	18		
	30	70	TIME NOT		4	12	20	
	20	80	RECORDED					
	10	90						
	5	95						

TEST FLUID: ETHYLENE GLYCOL			ANODE: GOLD PLATED					
REACTION INHIBITOR	% TEST FLUID	% ADDED WATER	SPACE BETWEEN ELECTRODES (INCHES), TIME (MINUTES) .005"	.010"	.015"	.020"	.040"	.080"
	100	0						
	90	10						
	80	20						
	70	30						
	60	40					NO REACTION	
NONE	50	50	NOT TESTED					
	40	60			95			
	30	70			55			
	20	80			44			
	10	90			36	60		
	5	95			30	45		
	100	0						
	90	10						
	80	20						
	70	30						
	60	40						
1g	50	50	NOT					
BZT	40	60	TESTED				NO REACTION	
	30	70						
	20	80						
	10	90						
	5	95						
TEST FLUID: ETHYLENE GLYCOL			ANODE: RHODIUM PLATED					
	100	0						
	90	10						
	80	20						
	70	30	NOT				NO REACTION	
	60	40	TESTED					
NONE	50	50			90			
	40	60			44	78		
	30	70			30	60		
	20	80			12	32		
	10	90			4	10		
	5	95			3	7		
	100	0						
	90	10						
	80	20						
	70	30					NO REACTION	
	60	40						
1g	50	50						
BZT	40	60	NOT					
	30	70	TESTED					
	20	80			32			
	10	90			25			
	5	95			12	30		

TEST FLUID: PROPYLENE GLYCOL			ANODE: SILVER PLATED					
REACTION INHIBITOR	% TEST FLUID	% ADDED WATER	SPACE BETWEEN ELECTRODES (INCHES), TIME (MINUTES) .005"	.010"	.015"	.020"	.040"	.080"
NONE	100	0	25	30				
	90	10		25		NO REACTION		
	80	20	1 - 10	20				
	70	30		15				
	60	40	1	1 - 10	12	20		
	50	50	LESS THAN			30		
	40	60	1 MINUTE			20	40	
	30	70					22	
	20	80					12	
	10	90			1 TO 10 MINUTES			
	5	95						14
½g BZT	100	0	22					
	90	10	25					
	80	20						
	70	30	20			NO REACTION		
	60	40						
	50	50	1					
	40	60	TO					
	30	70	10	30				
	20	80		21				
	10	90	< 1		1 TO 10			
	5	95			MINUTES		21	40
1g BZT	100	0	30					
	90	10						
	80	20						
	70	30				NO REACTION		
	60	40	30					
	50	50	16					
	40	60						
	30	70	1 TO 10	24				
	20	80		20				
	10	90	< 1	1 TO 1C MINUTES				
	5	95					24	60
2g BZT	100	0						
	90	10						
	80	20						
	70	30				NO REACTION		
	60	40						
	50	50						
	40	60						
	30	70	1 TO 10					
	20	80		15				
	10	90				20		
	5	95	< 1	1 TO 10 MINUTES			12	
5g BZT	100	0						
	90	10						
	80	20						
	70	30						
	60	40			NO REACTION			
	50	50						
	40	60						
	30	70						
	20	80						
	10	90	1					
	5	95		10				

TEST FLUID: PROPYLENE GLYCOL			ANODE: SILVER PLATED					
REACTION INHIBITOR	% TEST FLUID	% ADDED WATER	SPACE BETWEEN ELECTRODES (INCHES), TIME (MINUTES)					
			.005"	.010"	.015"	.020"	.040"	.080"
	100	0	50					
	90	10		50				
	80	20						
	70	30					NO REACTION	
	60	40			75			
2g EMI-24	50	50	TIME NOT					
	40	60	RECORDED					
	30	70				60		
	20	80						
	10	90					15	
	5	95					5	
	100	0						
	90	10	10					
	80	20	3					
	70	30		15			NO REACTION	
	60	40		4	18			
5g EMI-24	50	50			12			
	40	60			4			
	30	70	TIME NOT		35	20		
	20	80	RECORDED		5	2		
	10	90			LESS THAN 1			
	5	95						
	100	0	40					
	90	10						
	80	20						
1g	70	30						
BZT	60	40	45				NO REACTION	
&	50	50						
2g	40	60	1 TO 10					
EMI-24	30	70	MINUTES					
	20	80						
	10	90	1			8		
	5	95						
	100	0						
	90	10						
	80	20	4					
	70	30						
	60	40	< 1	4		NO REACTION		
1g TOT	50	50						
	40	60						
	30	70	TIME NOT		2			
	20	80	RECORDED			2		
	10	90					5	
	5	95						

TEST FLUID: DEICING FLUID MIL-A-8243				ANODE: SILVER PLATED					
REACTION INHIBITOR	% TEST FLUID	% ADDED WATER	% GLYCOLS	SPACE BETWEEN ELECTRODES (INCHES), TIME (MINUTES)					
				.005"	.010"	.015"	.020"	.040"	.080"
NONE	100	0	88						
	90	10	79						
	80	20	70						
	70	30	62		1 TO 10 MINUTES			NO REACTION	
	60	40	53	1					
	50	50	44						
	40	60	35					80	
	30	70	26	LESS THAN 1 MINUTE				60	
	20	80	18					45	
	10	90	9						
	5	95	4						
¼g BZT	100	0	88			15			
	90	10	79		20				
	80	20	70					NO REACTION	
	70	30	62						
	60	40	53						
	50	50	44						
	40	60	35	1 TO 10 MINUTES					
	30	70	26						
	20	80	18						
	10	90	9	LESS THAN 1 MINUTE					
	5	95	4						
1g BZT	100	0	88						
	90	10	79	1					
	80	20	70	TO					
	70	30	62	10				NO REACTION	
	60	40	53						
	50	50	44		12				
	40	60	35						
	30	70	26						
	20	80	18				1		
	10	90	9	LESS THAN 1 MINUTE				1 TO 10	
	5	95	4						
2g BZT	100	0	88						
	90	10	79						
	80	20	70						
	70	30	62	20				NO REACTION	
	60	40	53	11					
	50	50	44						
	40	60	35	1 TO 10 MINUTES		15			
	30	70	26						
	20	80	18				2		
	10	90	9						
	5	95	4	LESS THAN 1 MINUTE				1 TO 10	
5g BZT	100	0	88						
	90	10	79						
	80	20	70						
	70	30	62						
	60	40	53					NO REACTION	
	50	50	44						
	40	60	35						
	30	70	26						
	20	80	18			2			
	10	90	9	TIME NOT RECORDED			2		
	5	95	4					3	

TEST FLUID: DEICING FLUID MIL-A-8243				ANODE: SILVER PLATED					
REACTION INHIBITOR	% TEST FLUID	% ADDED WATER	% GLYCOLS	ELECTRODE SPACING (INCHES), TIME (MINUTES) .005"	.010"	.015"	.020"	.040"	.080"
	100	0	88						
	90	10	79	60					
	80	20	70		50				
	70	30	62					NO REACTION	
	60	40	53						
1g EMI-24	50	50	44			15			
	40	60	35						
	30	70	26	TIME NOT					
	20	80	18	RECORDED			1		
	10	90	9					7	
	5	95	4						
	100	0	88						
	90	10	79	50					
	80	20	70	15					
	70	30	62					NO REACTION	
	60	40	53	> 1	30				
2g EMI-24	50	50	44			60			
	40	60	35						
	30	70	26	TIME NOT		1			
	20	80	18	RECORDED					
	10	90	9				1		
	5	95	4						
	100	0	88						
	90	10	79						
	80	20	70	5					
	70	30	62						
	60	40	53	TIME			NO REACTION		
5g EMI-24	50	50	44	NOT					
	40	60	35	RECORDED					
	30	70	26						
	20	80	18		2				
	10	90	9		1				
	5	95	4						
	100	0	88	90					
	90	10	79	26					
	80	20	70						
	70	30	62	12			NO REACTION		
	60	40	53						
1g BZT & 2g EMI-24	50	50	44	1	30				
	40	60	35	TO	20				
	30	70	26	10	12				
	20	80	18		1 TO 10	1			
	10	90	9					1	
	5	95	4	LESS THAN 1 MINUTE			1 TO 10		
	100	0	88						
	90	10	79						
	80	20	70						
	70	30	62						
	60	40	53			NO REACTION			
5g BZT & 5g EMI-24	50	50	44						
	40	60	35						
	30	70	26						
	20	80	18	1	.30				
	10	90	9		> 1				
	5	95	4						

TEST FLUID: DEICING FLUID MIL-A-8243 — ANODE: SILVER PLATED

REACTION INHIBITOR	% TEST FLUID	% ADDED WATER	% GLYCOLS	.005"	.010"	.015"	.020"	.040"	.080"
				ELECTRODE SPACING (INCHES), TIME (MINUTES)					
1g TOT	100	0	88	75					
	90	10	79	65					
	80	20	70			NO REACTION			
	70	30	62						
	60	40	53						
	50	50	44		60				
	40	60	35			50			
	30	70	26						
	20	80	18					60	
	10	90	9		TIME NOT RECORDED				
	5	95	4						

TEST FLUID: DEICING FLUID MIL-A-8243 — ANODE: GOLD PLATED

REACTION INHIBITOR	% TEST FLUID	% ADDED WATER	% GLYCOLS	.005"	.010"	.015"	.020"	.040"	.080"
NONE	100	0	88	NOT TESTED	NOT TESTED			NO REACTION	
	90	10	79			100			
	80	20	70			70			
	70	30	62			55			
	60	40	53			48	70	95	
	50	50	44			45	60	70	
	40	60	35			40	45	60 (40 and 30)	
	30	70	26			32	20		
	20	80	18			19	25	40	
	10	90	9				15 (10 and 5)	30	
	5	95	4			10		22	
1g BZT	100	0	88	NOT TESTED	NOT TESTED			NO REACTION	
	90	10	79						
	80	20	70						
	70	30	62						
	60	40	53			78			
	50	50	44			55			
	40	60	35			45			
	30	70	26			60			
	20	80	18			36	60		
	10	90	9			30	38		
	5	95	4			25	8		

TEST FLUID: DEICING FLUID MIL-A-8243 — ANODE: RHODIUM PLATED

REACTION INHIBITOR	% TEST FLUID	% ADDED WATER	% GLYCOLS	.005"	.010"	.015"	.020"	.040"	.080"
NONE	100	0	88	NOT TESTED	NOT TESTED	58			NO REACTION
	90	10	79			35	40		
	80	20	70			15	18	28	
	70	30	62			10	13	15	
	60	40	53			8	10	13	
	50	50	44			4		16	
	40	60	35			2	8	13	
	30	70	26			3	5	12	
	20	80	18			2	7	8	
	10	90	9			3	6	10 (10 and 5)	
	5	95	4			2	3		
1g BZT	100	0	88	NOT TESTED	NOT TESTED			NO REACTION	
	90	10	79						
	80	20	70						
	70	30	62						
	60	40	53						
	50	50	44						
	40	60	35						
	30	70	26						
	20	80	18			13			
	10	90	9			4	18	35	
	5	95	4			2	6	15	

TEST FLUID: SYNTHETIC DEICING*				ANODE: SILVER PLATED					
REACTION INHIBITOR	% TEST FLUID	% ADDED WATER	% GLYCOLS	SPACE BETWEEN ELECTRODES (INCHES), TIME (MINUTES)					
				.005"	.010"	.015"	.020"	.040"	.080"
NONE	100	0	90	10	40				
	90	10	81	5	30	55	65		
	80	20	72			50		NO REACTION	
	70	30	63						
	60	40	54					80	
	50	50	45					20	
	40	60	36		TIME NOT RECORDED				
	30	70	27						
	20	80	18						
	10	90	9						50
	5	95	5						
½g BZT	100	0	90						
	90	10	81						
	80	20	72						
	70	30	63	40				NO REACTION	
	60	40	54						
	50	50	45		30				
	40	60	36			25			
	30	70	27				30		
	20	80	18	TIME NOT RECORDED				40	
	10	90	9						
	5	95	5						
1g BZT	100	0	90						
	90	10	81						
	80	20	72						
	70	30	63	60				NO REACTION	
	60	40	54	15					
	50	50	45	3					
	40	60	36		3				
	30	70	27			8			
	20	80	18	LESS THAN 1 MINUTE			75		
	10	90	9					30	
	5	95	5						
2g BZT	100	0	90						
	90	10	81						
	80	20	72						
	70	30	63				NO REACTION		
	60	40	54						
	50	50	45						
	40	60	36						
	30	70	27	10					
	20	80	18		18	40			
	10	90	9	TIME NOT RECORDED				35	
	5	95	5						

*SYNTHETIC DEICING FLUID IS 90% ETHYLENE GLYCOL AND PROPYLENE GLYCOL (3 TO 1 RATIO) AND 10% WATER

TEST FLUID: WATER BOILER FLUID* — **ANODE: SILVER PLATED**

REACTION INHIBITOR	% TEST FLUID	% ADDED WATER	% GLYCOLS	.005"	.010"	.015"	.020"	.040"	.080"
				SPACE BETWEEN ELECTRODES (INCHES), TIME (MINUTES)					
0.1g BZT	100	0	5	LESS THAN 1		1	5	20	
0.2g BZT				< 1		8			
0.3g BZT				< 1	3			NO REACTION	
0.4g BZT				< 1	5				
0.5g BZT				1					
1.0g BZT				2					
1.5g BZT									
2.0g BZT									
NO INHIBITORS					<1	1	2	6	15
1.0g BZT & 1.0g EMI-24				2					
0.5g TOT					4		6		
1.0g TOT					10	3			
1.5g TOT					5				
2.0g TOT									
5.0g TOT									

*WATER BOILER FLUID IS 5% PROPYLENE GLYCOL IN WATER

TEST FLUID: ETHYLENE GLYCOL — **ANODE: SILVER PLATED**

‡ 3g NaCl per 100 ml water

REACTION INHIBITOR	% TEST FLUID	% ADDED WATER	% ADDED SALT WATER‡	.005"	.010"	.015"	.020"	.040"	.080"
½g BZT	100	0	0	17	27				
	99		1	80					
	90	10		21	30		NO REACTION		
	90		10	25					
	80	20		34	40				
	80		20	< 1	3	100			
	50	50		1 TO 10		15	75		
	50		50	< 1					
	20	80		LESS THAN 1 MINUTE		1 TO 10		30	
	20		80						

TEST FLUID: PROPYLENE GLYCOL

REACTION INHIBITOR	% TEST FLUID	% ADDED WATER	% ADDED SALT WATER	.005"	.010"	.015"	.020"	.040"	.080"
½g BZT	100	0	0	22					
	99		1						
	90	10		75					
	90		10			NO REACTION			
	80	20							
	80		20						
	50	50		1 TO 10					
	50		50	<1					
	20	80		1 TO 10	20				
	20		80	<1	1 TO 10		14		

TEST FLUID: DEICING FLUID (MIL-A-8243)

REACTION INHIBITOR	% TEST FLUID	% ADDED WATER	% ADDED SALT WATER	.005"	.010"	.015"	.020"	.040"	.080"
½g BZT	100	0	0	1 TO 10		15			
	99		1	70					
	90	10		1 TO 10	20				
	90		10				NO REACTION		
	80	20		1 TO 10					
	80		20						
	50	50		1 TO 10 MINUTES					
	50		50	<1					
	20	80		LESS THAN 1 MINUTE					
	20		80						

Table III. Summary of Effective Reaction Inhibitor Levels for Various Glycol Solutions

Glycol Solution	Reaction Inhibitor (gms/100 ml Solution)	Range of Solution Concentration for Reaction Inhibiting
Ethylene Glycol (100%)**	1g BZT 2g BZT	50% to 100%* 30% to 100%
Propylene Glycol (100%)	None	70% to 100%
Propylene Glycol (5%) (Water boiler fluid)	0.5g BZT	0 to 100%
Deicing Fluid MIL-A-8243B	1g BZT 1g TOT	50% to 100% ǂ 50% to 100% ǂ

* Normal usage concentration 62%.

** This is without corrosion inhibitors or other additives. Reaction tests needed when additives are defined.

ǂ This includes the dilution from rain and snow. Often this fluid is diluted prior to application to concentrations as low as 20%. When the fluid is to be diluted, additional reaction inhibitor should be added at the time of dilution.

For solution concentrations less than the reaction inhibiting range indicated in this Table, inspection and maintenance of electrical system integrity should be relied upon for hazard elimination rather than the use of excessive amounts of BZT reaction inhibitor.

VIII. Glycol Penetration and Immersion Tests

Concurrent with glycol reaction tests and reaction inhibitor tests, other tests were performed to determine the susceptibility of electrical connectors and electrical wiring to penetration or material degradation when exposed to glycol solutions. These tests consisted of altitude cycling of immersed connectors, soak tests of mated connectors, and soak tests of wires and unmated connectors.

A. Altitude Cycling of Immersed Connectors

Twelve connectors were tested:

4 - MS24266R14B4SN
4 - MS24266R24B43S
4 - MS3126E22B55S

The first two connector types were chosen because failures had occurred with these types. The third connector type was chosen to permit testing with a different connector design. These plugs were wired to connect alternate contacts to opposite polarities of the 28 volt dc power source. Fillers were installed in unused insert holes. The plugs were mated to their receptacles and mounted vertically with plugs submerged in a 50% propylene glycol/water solution. The assembly was placed in an altitude chamber and energized with 28 volts dc during the altitude tests. Leakage current was monitored throughout the test with a 0.02 ma sensitivity meter.

The altitude chamber pressure was reduced to approximately 1.0 inches of mercury and maintained at this pressure for 30 minutes. Chamber pressure was then returned to ambient pressure within 1 minute and maintained at ambient pressure for 30 minutes. These tests were repeated for three consecutive cycles. Circuit conductivity was checked with a megohmmeter after each cycle.

There was no evidence of conduction in any of the 12 mated pairs of connectors during or after the altitude cycling tests. The shell was removed from one of each type of connector. There was no evidence of degradation of materials. The removed inserts were tested for absorbed glycol or the leakage of glycol beyond the wire seal. No glycol was detected.

B. Soak Tests of Mated Connectors

Three plugs of each type used in the altitude cycling test were wired and mated with the proper receptacles. One set of a mated pair of each type was submerged in a 50% ethylene glycol/water solution. Another set was submerged in a 50% propylene glycol/water solution. The third set was submerged in a 50% deicing fluid/water solution. The connectors were periodically measured for insulation resistance as evidence of glycol/water penetration.

The connectors were soaked in the glycol solution for a total of 43 days. There was no evidence of glycol penetration. All circuits were isolated by more than 2×10^9 ohms.

C. Soak Tests of Wires and Unmated Connectors

Nine unmated plug type connectors taken from the altitude cycling test were soaked in the same three fluids used in the mated connector soak tests. Samples of Number 12, 16, 18, 20, 22, and 24 gage polyalkene and kynar insulated wires were completely submerged and soaked in the same three fluids.

Conductivity of the wire insulation was measured periodically. The connectors were periodically removed from the solutions, external surfaces were dried and conductivity measured. After 20 days, the connectors were transferred to connector internal exposure tests.

Wire samples were soaked for a total of 71 days in the glycol solutions. There was no evidence of conduction or of physical change. Insulation resistance of three foot samples was greater than 2×10^{12} ohms.

After 20 days in the glycol solutions unmated, most of the connectors had lower insulation resistance but all measurements were too high to be considered conductive. The minimum reading was one megohm for the MS3126 connectors and 30 megohms for the MS24266 connectors.

IX. Induced Electrical Connector Failures

The glycol immersion tests of Section VIII showed no tendency of connectors or wiring to deteriorate or establish reaction paths when exposed to glycol solutions. Exploratory tests had shown that damaged wiring at the connector could be used to start a glycol reaction with the resulting fire propagating into the connector. Tests were performed to further identify the failure mechanism. These tests included electrolysis of sectioned connectors, tests of damaged connectors, and internal exposure of connectors to glycol solutions.

A. Electrolysis of Sectioned Connectors

The relative activity of the metals used in connectors was compared by observing the formation of reaction products. Two test specimens were prepared by sectioning connector assemblies and placing wired contacts along the section. Contact pairs were energized at 28 volts dc and four test solutions were applied between contacts. The test solutions were 10% ethylene glycol, 10% propylene glycol, 10% deicing fluid, and demineralized water. Tests were conducted with size 20 rhodium plated sockets in an MS24266 connector and size 20 gold plated sockets in an MS3126 connector.

In the MS24266 connector, the most active area was found to be the crimp barrel in the deformed areas. The beryllium contact retainer clips were second in activity. Uncrimped rhodium plated areas were the least active. The silver plated wire was active when exposed but it is normally insulated in the assembly and was not active.

In the MS3126 connector, the most active area was the crimp barrel. Next in activity was the uncrimped gold plated areas. The rhodium plated pin barrel was the least active.

The most active solution was found to be ethylene glycol, followed by propylene glycol, deicing fluid, and water. In other tests, the 10% concentration was found to be among the most active with higher concentrations less active.

Each reaction was allowed to proceed until the solution was electrolized. The relative electrochemical activity

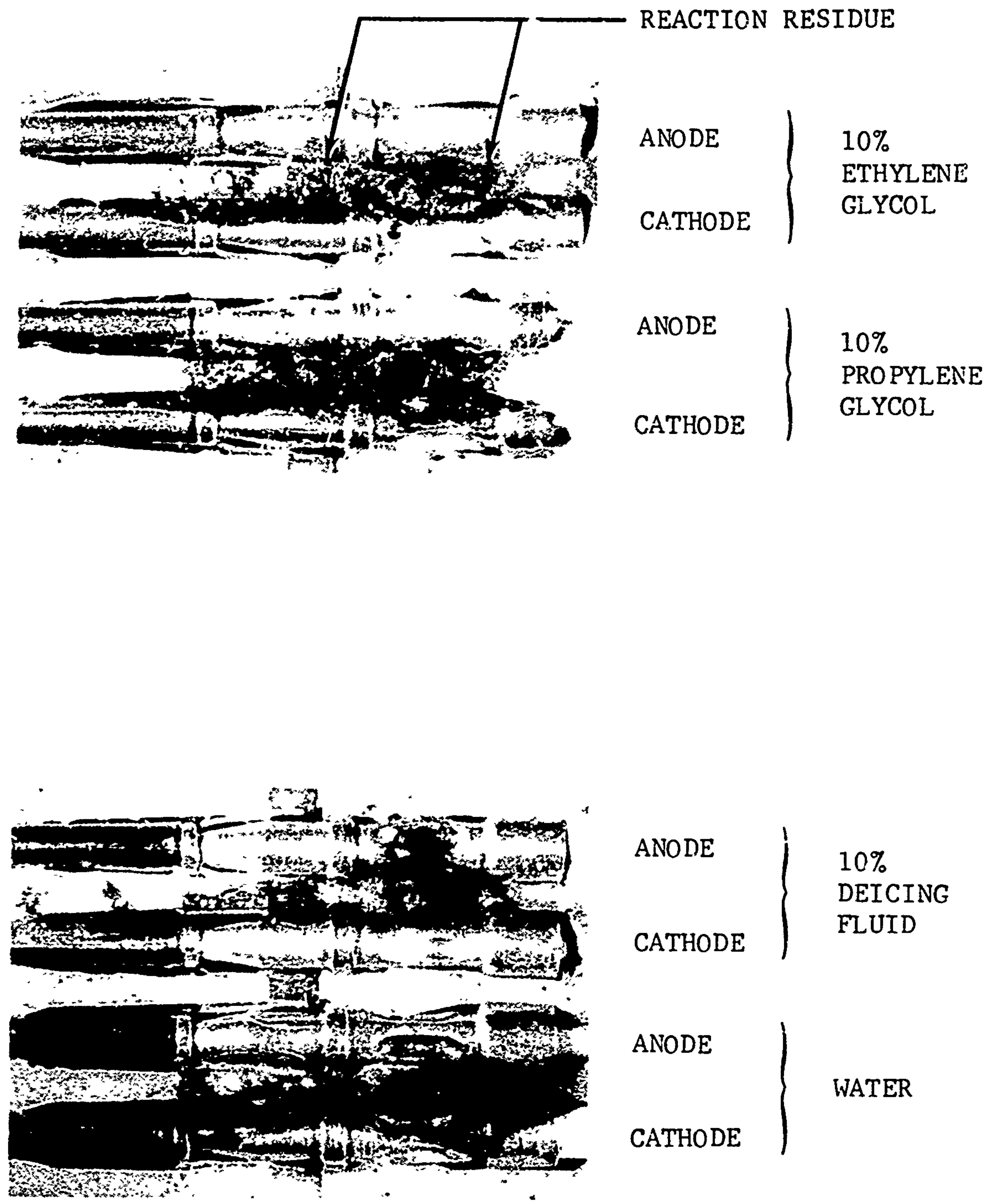


Fig. 4 GLYCOL REACTION AFTER MS24266 CONNECTOR WAS SECTIONED

was roughly proportional to the quantity of residue remaining in each area.

In similar tests, heavy residues were formed by repeated applications of solution. The location of reaction products is shown in Figure 4. In each case, the residue produced arcing and radio noise and caused some pitting and local burning of the rubber surface. The burned rubber formed a conductive powder but did not maintain a circuit along the open surface.

Reaction in ac circuits was compared by applying 115 volts 400 Hz to a pair of socket contacts. The solution was dissipated rapidly but no reaction products were visible.

B. Damaged Connector Tests

Several connectors were intentionally damaged and glycol solutions were added between energized circuits to observe the extent of damage produced. A 10% ethylene glycol/water solution and several other glycol solutions were used. Several damaged configurations were tested:

(1) Insert Damage

The rubber insert was deliberately damaged at the wire seal by improper use of insertion tools to produce a cut between adjacent contacts or between contacts and the shell. Repeated application of the glycol solution over a period of several days produced the usual reaction products. At 28 volts, the conductivity decreased as the solution was electrolyzed and in all cases conductivity stopped with no damage.

(2) Conductor Exposure

Tests were conducted with conductors exposed at the surface of the rubber insert. Exposure was accomplished by over stripping the wire and by opening the wire seal by inserting filler plugs beside the wire and leaving them in place. The solution was applied for several days and reaction products formed across the surface. Conductivity stopped with no damage.

It was found that the insert could be damaged by applying higher voltages (200 to 400 volts, depending on spacing) or by reducing the spacing to about .040 inches at 28 volts. At the actual spacing and voltage of the connectors, no damaging reaction was produced.

(3) Combined Damage

With a combination of insert damage and overstripping, sustained conduction was obtained at 28 volts with full spacing. A reaction was established in a cut between two wires that were exposed by overstripping. After building reaction products and reaching a drying-out stage, the residue produced sufficient arcing to burn the surface of the rubber insert material within the cut. The burned rubber formed a conductive powder and since the powder was confined a conductive path was maintained. The burn progressed the full length of the involved contacts and the contact was brought to a red heat without tripping a 20 ampere circuit breaker.

C. Connector Internal Exposure to Glycol Solutions

The effect of trapping glycol solutions inside the connectors was investigated initially in conjunction with the altitude test in order to determine the probable result of leakage. An unused pair of connectors type MS24264R16B24S and MS24266R16B24P, were wired with six parallel circuits and were mated after being filled with a 50 percent ethylene glycol solution. The resistance between circuits was 8 megohms after mating. The connector was energized for 3 days at 28 volts with no evidence of conduction or damage. Two weeks later the resistance was again checked and was found to be about 1000 ohms. It was then connected to a 28 volt line and soon became conductive with rapid heating. The test was interrupted to minimize the damage and the specimen was sectioned and examined.

The test was repeated using one connector of each type with one of three different glycol solutions (a total of nine plugs and receptacles). The glycol solutions used were:

(1) 10% ethylene glycol/water solution

(2) 10% propylene glycol/water solution

(3) 10% deicing fluid/water solution.

The nine plugs had been used in the soak test. The nine receptacles had been used in the altitude cycling test and had no previous internal expcsure to glycol. Each connector pair was filled with one of the solutions, mated, and mounted with the plug up. The connectors were energized with 28 volts dc for 8 hours a day. Total conductivity of the nine specimens was 200 microamperes at 28 volts. Conductivity decreased with time.

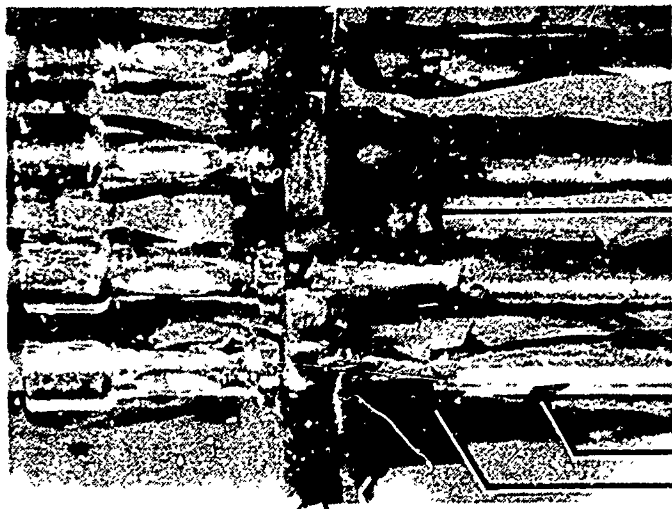
After two days, the MS24264R14B4P specimen with ethylene glycol became conductive and heated rapidly. A size 24 test lead was fused by the current but a 20 ampere circuit breaker did not trip. The mating plug had been soaked for 20 days but remained clear and was not damaged since the small lead interrupted the current. The failed specimen was sectioned and examined. The breakdown was found to along the solid retainer on the mating side. The retainer was burned across the entire area between the positive and negative pins. The rubber insert was burned to almost the full diameter of the connector.

After four days of internal exposure, the MS24266R24B43S specimen with ethylene glycol suddenly became conductive. It expelled three filler plugs, heated rapidly, smoked, and in about 10 seconds had fused the size 24 test lead without tripping a 20 ampere circuit breaker. The specimen was sectioned and the breakdown was found to be at the solid retainer, primarily on the wire side.

Figure 5 shows the breakdown area inside the connectors. The glycol solution apparently reached the insulating connector retainer and bridged between 2 or more connections.

The remaining seven specimens were energized for 52 days total test time with no breakdown. After 42 days, the conductivity of the seven connectors had decreased from 0.25 ma to 0.05 ma at 28 volts dc. At that time, each connector was unmated, examined, filled again, mated, and energized for the remaining 10 days. There was no breakdown or evidence of deterioration.

MS24266R16B24S
CONNECTOR



MS24264R14B4P
CONNECTOR

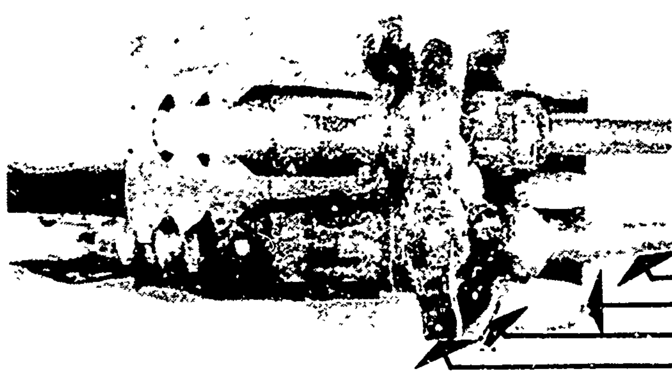


Fig. 5 CONNECTOR DAMAGE AFTER FILLING WITH GLYCOL, MATING, AND ENERGIZING

These tests showed that connectors may react to internal exposure to glycol solutions. However, many more tests would be required to establish which glycol solution is most likely to penetrate to the contacts, which type connector is most susceptible to penetration, and the probability of connector failure from internally introduced glycols.

X. Glycol Contamination Detection

Investigations of possible methods of detecting that airplane areas had been contaminated with glycol solutions were made as part of the hazard reduction program.

Two methods of detecting the presence of glycols on a wetted surface were evaluated: ultraviolet radiation and infrared spectrophotometry. Attempts to detect glycol contamination by ultraviolet radiation were unsuccessful. Infrared spectophotometric examination of extracted wash fluid detected the presence of 1 mg of glycol. However, this method is of limited value because it is restricted to the laboratory.

Some areas of an airplane are deliberately sprayed with deicing fluid and cannot be considered contaminated unless equipment and components were not properly protected. Any surface that is not obviously wet can be considered free from glycol reaction hazards. Wet surfaces suspected of glycol contamination can be cleaned by the simple procedure of Section XI.

XI. Glycol Decontamination

Two methods were tested for decontaminating areas wetted with glycol solutions: water rinsing, and reaction inhibiting fluid rinsing. The objective in cleaning an area by water rinsing was to reduce the glycol concentration below 1% as a minimum and preferably to completely dry the area. The objective of the reaction inhibiting fluid rinse was to introduce enough reaction inhibitor to the area to prevent a glycol/metal electrolytic reaction. It should be noted that the requirement was to prevent a glycol reaction. The decontamination problem studied by NASA (Reference 3) also involved removing certain corrosion inhibitors which became corrosive when exposed to the atmosphere.

Both the water rinse and the reaction inhibitor fluid rinse satisfactorily decontaminated the area. The use of a reaction inhibitor rinse is not recommended. The water rinse was completely satisfactory. A reaction inhibitor rinse introduces other chemicals to the area unnecessarily.

XII. Reaction Inhibitor Detection

In the event that there is a need to determine the quantity of benzotriazole in a glycol solution, the following procedure is recommended (Reference 9):

Reagents:

Silver nitrate solution, 10 g per 100 ml.
Ammonium Hydroxide, sp. gr. 0.88
EDTA solution - Add 40 g of disodium dihydrogen ethylene - diaminetetra - acetate to 50 ml of water. Add ammonium hydroxide solution to dissolve the salt. Dilute the solution to 100 ml with water.

Procedure:

Place 10 ml of EDTA solution, 5 ml of silver nitrate solution and 10 ml of water in a 250 ml beaker. Heat to about 50°

Add 7 ml of ammonia solution and 50 ml of sample. Heat to $90^{\circ}C$ and maintain for 15 minutes.

Cool solution and filter through a weighed, sintered-glass crucible.

Wash the precipitate with six 10 ml portions of water and dry it at $120^{\circ}C$ for 15 minutes.

Cool the crucible and weigh.

Calculations:

g BZT per 100 ml = wt of ppt X 1.05

XIII. Reaction Inhibitor Characteristics

A. Materials Compatibility

The aircraft materials listed in Table IV were tested for compatibility with deicing fluid and with deicing fluid with 1 gram of benzotriazole per 100 milliliters of deicing fluid. The materials were weighed and then immersed in the test fluids for one month at room temperature. The materials were then washed, dried, re-weighed, and examined for evidence of corrosion or other visible effects. There was no evidence of incompatibility.

B. Solubility

Reaction tests established the effects of various quantities of reaction inhibitors on the glycol/metal reactions. With these quantities established, it was necessary to determine if these quantities would remain in solution at low temperatures. These tests are summarized in Table V.

C. Benzotriazole Storage Stability

A series of reaction tests were conducted to determine if benzotriazole changed its reaction inhibiting properties after storage. A solution of 1 gram of BZT per 100 ml ethylene glycol was mixed and its reaction characteristics with silver plated wire electrodes were obtained. The solution was tested in the same manner at one month intervals for a total storage time of 5 months. No significant change in reaction inhibiting characteristics was obtained.

D. Toxicity of Benzotriazole

The health hazards of benzotriazole have been evaluated by several investigators. The U. S. Department of Health, Education, and Welfare Toxic Substance List, 1972 Edition gives the lethal dose as 1000 mg per 1000 g body weight. Sherwin Williams Chemical Company reports the LD_{50} value in white rats as 675 mg/kg. These values are considered to be in the low to moderate toxicity range. Dermal toxicity studies show benzotriazole to be non-toxic and non-irritating to the skin.

Table IV. Materials Compatibility

Material	Finish	MIL-A-8243B fluid: Wt. change mg/in^2	MIL-A-8243B fluid: Corrosion or other effect	1g B7T in 100 ml MIL-A-8243B fluid: Wt. change mg/in^2	1g B7T in 100 ml MIL-A-8243B fluid: Corrosion or other effect
Aluminum alloys					
356-T6/-T71 Casting	MIL-A-8625 - Type II	0	none	0	none
SC114A Die Casting	Hard anodize	0	none	0	none
2024 T-4	MIL-A-8625 Type II	0	none	0	none
5052-0	-	0	none	0	none
6061-T6	-	0	none	0	none
6061-T651	MIL-A-8625 Type II	0	none	0	none
7075-T6	Hard anodize	0	none	0	none
Steels					
17-7 PH	Passivated	0	none	0	none
304 Cres		0	none	0	none
303 Cres	Passivated	0	none	0	none
302 Cres	Passivated	0	none	0	none
Non-Metals					
Fluorosilicone (LS-53)		+0.031	none	+0.032	none
Teflon		0	none	0	none
Buna-N		0	none	0	none
Nylon Type I		0	none	0	none
Parker Hannifen "Duraflote"		not tested	none	+0.018	none

Table V. Low Temperature Tests on Reaction Inhibited Glycols

Test Fluid Type	Test Fluid %	Reaction Inhibitor grams/100 ml of Test Fluid	% Added Water	Temperature °F (4) Solution Clear (5)	Temperature °F (4) Frozen (6)
Ethylene Glycol	100	0	0	-50	-65,-80
	50	0	50	-40	-50,-80
	10	0	90	+25	+17,-80
	100	2 BZT	0	-50	-65,-80
	50	2 BZT	50	-40	-50,-80
	10	2 BZT	90	+25	+17,-80
	100	5 BZT	0	-50	-65,-80
	50	5 BZT	50	-30	-40,-80
	10	5 BZT	90	(1)	+25,-80
Propylene Glycol	100	0	0	-50	-65,-80
	50	0	50	-40	-50,-80
	10	0	90	+25	+17,-80
	100	2 BZT	0	-50	-65,-80
	50	2 BZT	50	-30	-40,-80
	10	2 BZT	90	+25	+17,-80
	100	5 BZT	0	-50	-65,-80
	50	5 BZT	50	-10	-20,-80
	10	5 BZT	90	+25	+17,-80
Deicing Fluid MIL-A-8243B	100	0	0	-50	-65,-80
	50	0	50	-10	-20,-80
	10	0	90	+25	+17,-80
	100	2 BZT	0	-50	-65,-80
	50	2 BZT	50	-10	-20,-80
	10	2 BZT	90	+25	+17,-80
	100	5 BZT	0	-50	-65,-80
	50	5 BZT	50	-10	-20,-80
	10	5 BZT	90	+25	+17,-80
	100	2 EMI-24	0	-50	-65,-80
	50	2 EMI-24	50	-10	-20,-80
	10	2 EMI-24	90	+25	+17,-80
Water Boiler Fluid (5% Propylene Glycol in Water)	100	0	0	+25	+17,-80
	100	0.5 BZT	0	+25	+17,-80
	100	1.0 BZT	0	+25	+17,-80
	100	1.5 BZT	0	+25	(3)
	100	2.0 BZT	0	(2)	(3)

NOTES:

1. Semi-frozen at +25°F.
2. Insoluble residue at +25°F (solution clear at room temperature).
3. Frozen at +17°F to -80°F test points, thaws with an insoluble residue.
4. Test temperatures 25, 17, 0, -10, -20, -30, -40, -50, -65, and -80°F.
5. "Solution Clear" temperature is the minimum test temperature at which the solution remained a clear liquid.
6. Freeze temperatures are the range of test points over which the solution was frozen but on warming thawed to a clear liquid.

Benzotriazole is similar in chemical structure to the material aminotriazole which is known to have carcenogenic properties. The National Cancer Society is conducting studies on the carcenogenic properties of benzotriazole. Tests are expected to be completed by mid-1975.

The Occupational Safety and Health Administration of the U. S. Department of Labor (OSHA) has been contacted for information on federal regulations of benzotriazole. They stated that at the time of inquiry (August 1974) there were no applicable federal standards for benzotriazole, ethylene glycol, or propylene glycol.

E. Flammability of Benzotriazole

The flash point of benzotriazole is 412°F. The material is considered to be non-flammable and the flammability of glycol solutions would not be effected by the addition of benzotriazole.

F. Environmental Impact

Since reaction inhibited fluid might be discarded in public drains and sewer lines, the eccological effect of the reaction inhibitor was checked. Reference 10 determined that benzotriazole would not adversely effect wastewater treatment facilities and that the material exhibits very low toxicity to trout, bluegills, and minnows. The Environmental Protection Agency (EPA) has no specific regulations on benzotriazole but this agency would have to approve its use based on the volume involved, and the frequency and season of its use.

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